NASA TECHNICAL NOTE

NASA TN D-5313

NASA TN D-5313

GROWTH OF 2H SILICON CARBIDE CRYSTALS

by J. Anthony Powell

Lewis Research Center

Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • JULY 1969

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ABSTRACT

Crystals of one of the rarer crystalline modifications of silicon, 2H SiC, were grown. These crystals with dimensions up to 3.3 mm in the c-direction and 0.3 mm in the a-directions were usually transparent and either colorless or amber colored. Growth was by the reduction of methyltrichlorosilane (CH_3SiCl_3) in a H_2 atmosphere in the temperature range 1300° to 1400° C. The vapor-liquid-solid (VLS) mechanism, which has been suggested for previous 2H SiC growth, was not involved in this growth. Factors affecting the crystal growth were the susceptor temperature profile and the CH_3SiCl_3 concentration in H_2.

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SUMMARY

Crystals of one of the rarer crystalline modifications of silicon carbide, 2H SiC, were grown. These crystals, with dimensions up to 3.3 millimeters in the c-direction and 0.3 millimeter in the a-directions, were usually transparent and either colorless or amber colored. Growth was by the reduction of methyltrichlorosilane (CH_3SiCl_3) in a hydrogen (H_2) atmosphere in the temperature range 1300° to 1400° C. Visual observations during growth showed that the crystal growth mechanism was not the vapor-liquid-solid (VLS) mechanism, a mechanism that has been proposed by others who have grown 2H SiC. Factors affecting the growth were the susceptor temperature profile and the CH_3SiCl_3 concentration in H_2.

The crystals grew on a vertical, cylindrically shaped, graphite susceptor which was inductively heated. The most successful 2H SiC growth occurred with a temperature of 1200° C at the top of the susceptor increasing to about 1500° C at the bottom. As the gas flowed down over the susceptor, two bands of growth occurred around the susceptor. At the top was a band of free silicon and polycrystalline β SiC, and below this at 1300° to 1400° C was a band of both 2H and β SiC crystals.

The CH_3SiCl_3 concentration in the H_2 affected the percentage of the 2H SiC crystals to the β SiC crystals. The percentage of 2H SiC increased with concentration in the range 0.4 to 0.8 molar percent. Below 0.4 molar percent the crystals were mostly β SiC; above 0.8 molar percent the 2H SiC crystals were heavily overgrown with polycrystalline β SiC.

INTRODUCTION

The maximum operating temperature of present semiconductor devices imposes serious design limitations in certain spacecraft and supersonic aircraft applications. An example is the weight penalty paid in cooling a power conditioning system in a high-temperature environment. Devices capable of higher operating temperatures would allow

more design flexibility where high-temperature environments are anticipated.

A semiconductor with much potential for applications where the ambient temperature is in excess of 400° C is SiC (ref. 1). This compound is grown in many different crystalline modifications called polytypes (ref. 2, pp. 107-108). The physical and chemical stability of all these polytypes is excellent to temperatures above 1000° C. The room-temperature energy band gaps for the SiC polytypes vary from about 2.3 to 3.2 electron volts. Theoretically, these band gaps should allow device operation in excess of 600° C. The highest measured values of the room-temperature electron mobility for the more common polytypes are comparable with silicon (refs. 3 and 4). Thermal conductivity, an important property in power devices, is greater for SiC than for most other semiconductors (ref. 5).

A major problem with SiC is the difficulty in growing single crystals of sufficient purity and perfection for the fabrication of semiconductor devices. No satisfactory process has yet been developed. The most common method for growing SiC to date has been the sublimation process (refs. 6 to 8). In this process, crystals grow from SiC vapor inside a hollow cavity in polycrystalline SiC. The temperature in the cavity is usually in the range 2300° to 2800° C. At these temperatures, SiC sublimes and then condenses on the cooler parts of the cavity. The result is uncontrolled nucleation. Under optimum conditions, clear hexagonal-shaped platelets of SiC have been grown. High-purity conditions are difficult to achieve at these temperatures, and the resulting crystals are often heavily doped. Also, the crystal platelets almost always have a layered structure of different polytypes.

The 2H polytype of SiC can be grown at temperatures less than 1500° C (refs. 9 to 15). This much lower growth temperature offers obvious advantages over the conventional sublimation process. However, up to this time, not much work has been done to determine the actual crystal growth mechanism and the optimum conditions for the growth of 2H SiC. In an attempt to get this information, crystals of 2H SiC were grown at Lewis. This report describes factors which affected this crystal growth and presents evidence that the vapor-liquid-solid (VLS) mechanism was not involved.

DISCUSSION OF THE CRYSTAL STRUCTURE AND SOME PROPERTIES OF SILICON CARBIDE

At this point, some discussion of the crystal structure of SiC is in order. In SiC every atom is at the center of a tetrahedron formed by four atoms of the other species. These tetrahedra are arranged so that parallel double layers of atoms are formed. Each double layer consists of a layer of silicon atoms in a hexagonal network parallel to a layer of carbon atoms also in a hexagonal network. Each silicon atom lies directly above

a carbon atom in the double layer. These double layers are stacked in a close-packed arrangement; therefore, only three relative positions of these double layers are possible. These positions are arbitarily designated A, B, and C. The stacking sequence of these double layers (A, B, and C) determines the particular crystalline modification. All stacking sequences can be described by the usual hexagonal axis system with one c-axis perpendicular to three equivalent axes each of which forms a 120° angle with each of the others. Any of the three equivalent axes can be designated as the a-axis. The stacking sequence thus refers to the order of the A, B, and C double layers along one dimension, the c-axis.

Since, by definition, one-dimensional polymorphism is called polytypism, these polymorphs of SiC are called polytypes. More than 40 different SiC polytypes have been discovered (ref. 2, pp. 107-108). The various stacking sequences in SiC result in one cubic structure and many hexagonal and rhombohedral structures. The sequence ABC gives the cubic structure known as β SiC. All other known stacking sequences give either a hexagonal or a rhombohedral structure and are generally known as α SiC. One of these, the sequence AB, gives the simplest hexagonal structure in SiC and is designated as 2H SiC. The most common polytype grown in the sublimation process is 6H SiC, which is also hexagonal and has the sequence ABCACB. A more complete discussion of the crystal structure of SiC is given in reference 2.

A semiconductor property of major importance in high-temperature applications is the energy band gap. In SiC, the magnitude of the room-temperature energy band gap ranges from a minimum for β SiC (about 2.3 eV) to a maximum for 2H SiC (about 3.2 eV). As a result, devices made from 2H SiC should be capable of operating at the highest temperature of all the known SiC polytypes.

Another important semiconductor property is the electron mobility which, in SiC, varies with polytype. A room-temperature electron mobility as high as 1000 square centimeters per volt per second has been measured in β SiC (ref. 5). Values of 300 and 500 square centimeters per volt per second have been measured in 6H and 15R SiC, respectively (ref. 4). The mechanism that limits electron mobility in SiC crystals from room temperature to 500° C is probably the type of scattering known as intervalley scattering (ref. 16). The symmetry of β SiC and 2H SiC results in selection rules for intervalley scattering more restrictive than those for the 6H and 15R polytypes (ref. 17). This appears to be the reason for the higher measured electron mobility in β SiC. Although the electron mobility has not been measured for 2H SiC, the same theoretical reason supports the belief that it will be higher than the other α SiC polytypes.

PREVIOUS CRYSTAL GROWTH OF 2H SILICON CARBIDE

Merz and Adamsky (ref. 9) first reported the growth of 2H SiC in 1959; since then

others (refs. 12 to 15) have also grown 2H SiC. Most of these crystals have been produced by the reduction of methyltrichlorosilane (CH_3SiCl_3) in an H_2 atmosphere at temperatures in the range 1350° C to 1525° C. Molar concentrations of CH_3SiCl_3 in H_2 from 0.1 to 0.8 percent were used. In most cases, the crystals grew on graphite substrates. Polycrystalline growth of β SiC is usually found growing next to, or in contact with, the 2H SiC crystals. The 2H SiC crystals grew in the form of small whiskers up to 3 millimeters long and up to 0.3 millimeter in diameter. The fast growth direction was along the c-axis.

The 2H SiC crystals grown by Merz and Adamsky grew in clusters as long thin prisms. Most of the crystals had globular masses of polycrystalline β SiC on the ends. Their crystals were mainly transparent and green, blue, or colorless.

The crystals grown by Patrick et al. (ref. 13) were usually colorless and tapered. The smaller crystals grew in clusters while the largest crystals always grew singly. The vapor deposition of metallic spots onto the graphite substrate usually increased the number of clusters, but the size of the crystals dropped. The occurrence of ball-like deposits attached to the crystals was observed. When the ball was attached to the middle of a whisker, it was found that the whisker was continuous through the ball, usually without any change in diameter. The whiskers almost always ended in a fine point free of any ball deposit. It was determined that the ball deposit was mostly β SiC, but the measurements made did not rule out the possibility of a small excess of silicon.

The 2H SiC whiskers grown by Ryan et al. (ref. 14) were similar to those just discussed. They found that if the graphite susceptor and the CH_3SiCl_3 were carefully purified, whisker growth would not occur. However, when they seeded the susceptor with certain metallic impurities, whiskers of SiC would grow on the susceptor. Most of these whiskers were 2H SiC. They also reported β SiC deposits on their 2H SiC whiskers.

THE VAPOR-LIQUID-SOLID (VLS) MECHANISM

Reports on the growth of 2H SiC so far have been somewhat conflicting as to the actual crystal growth mechanism. Wagner and Ellis (refs. 18 and 19) have suggested the vapor-liquid-solid (VLS) mechanism to explain the growth of 2H SiC. This mechanism was given further supporting evidence by Ryan et al. (ref. 14).

In the VLS mechanism, a whisker nucleates in a liquid droplet of some impurity on the graphite substrate. As the whisker grows, it emerges from the original droplet and carries some of the liquid along on the growing end. In this case the liquid droplet is a metallic solvent for SiC and also a preferred site for deposition of SiC from the vapor. The SiC supersaturates the liquid droplet and precipitates out on the growing whisker. Growth ceases when the liquid evaporates or is incorporated into the growing crystal.

Most of the 2H whiskers grown so far have been terminated by ball-shaped deposits, which are characteristic of VLS crystals. However, these ball-shaped deposits often are β SiC, and not a solidified solvent which would indicate VLS growth. From their observations, Patrick et al. (ref. 13) concluded that the β SiC attached to their 2H SiC whiskers was incidental to the whisker growth. Even though the addition of impurities, which might serve as possible solvents, increased the number of whiskers, they concluded that the VLS mechanism did not appear to provide an explanation for their 2H SiC whisker growth. Ryan et al. (ref. 14) observed that whiskers nucleated only when impurities were added to their graphite susceptors. They concluded that their 2H SiC growth took place by means of the VLS mechanism. Knippenberg and Verspui (ref. 15) demonstrated the presence of metallic solvents on the end of VLS grown crystals of β SiC and some α SiC polytypes. Although they grew 2H crystals in the presence of impurities, it was not clear whether the VLS mechanism was involved in this case.

From the preceding discussion, it can be stated that the question of the crystal growth mechanism for 2H SiC has not been settled. The results reported herein, however, establish that the VLS mechanism is not responsible for the 2H crystals grown at Lewis.

APPARATUS AND PROCEDURE

The procedure used in this work was similar to that used by others (refs. 11 and 13) in the growth of 2H SiC. Hydrogen was passed over liquid CH_3SiCl_3 in a saturator. This H_2-CH_3SiCl_3 vapor mixture was further diluted with more H_2, then passed through a mixing chamber and over an inductively heated susceptor inside a quartz reaction tube. Crystals of 2H SiC grew on the susceptor with specific susceptor temperature profiles and CH_3SiCl_3/H_2 concentrations.

The basic system (fig. 1) was all glass downstream from the flowmeters, except for a vacuum pressure gage and a vacuum thermocouple gage. These gages could be valved off with a vacuum stopcock. Viton O-rings were used to seal the various glass sections. Glass needle valves with Teflon stems and Viton O-ring seals were used on the saturator. The other valves in the system were standard vacuum stopcocks lubricated with silicone vacuum grease. Most of the glass tubing was 15-millimeter-inside-diameter pyrex. Upstream from the flowmeters, the system was stainless steel. Ultra-high-purity ($<$5 ppm N_2, 1 ppm O_2) hydrogen was used. The system could be evacuated and outgassed to less than 1 millitorr by means of a diffusion pump.

The saturator was constructed from a 300-cubic-centimeter flask. Hydrogen entering the saturator was directed over the surface of the liquid CH_3SiCl_3. The saturator was in a bath of carbon tetrachloride (CCl_4), which was maintained at a constant temper-

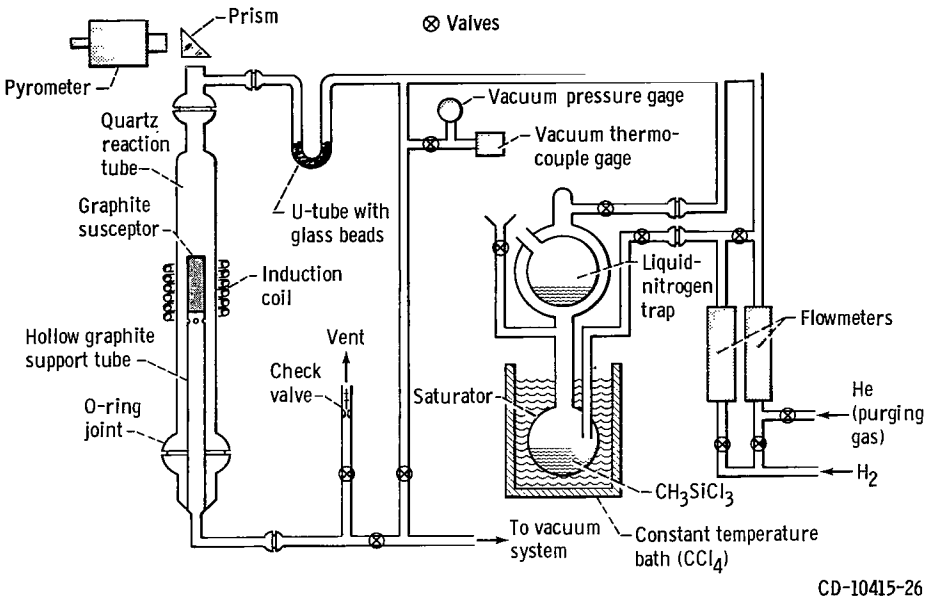

Figure 1. - Crystal growth system.

ature (±0.1° C) by means of a cooling coil also submerged in the CCl_4 bath. The H_2 leaving the saturator was assumed to be saturated with CH_3SiCl_3; thus, when the saturator temperature and H_2 flow rate were known, the CH_3SiCl_3 in H_2 concentration could be calculated. Hydrogen flow rates through the saturator ranged from 20 to 60 cubic centimeters per minute, saturator temperatures from -10° C to 6° C, and total H_2 flow rates from 200 to 600 cubic centimeters per minute. Concentrations of CH_3SiCl_3 in H_2 from 0.1 to 0.9 molar percent were used.

The following procedure was used to purify the CH_3SiCl_3. Commercial CH_3SiCl_3 of about 98 percent purity was obtained and fractionally distilled. This CH_3SiCl_3 was then placed in the saturator and vacuum distilled onto the liquid-nitrogen trap attached to the top of the saturator. This helped get rid of dissolved nitrogen and possibly other foreign gases.

A U-tube with 3-millimeter-diameter glass beads provided thorough mixing of the $CH_3SiCl_3 + H_2$ before it entered the reaction tube. A pyrex connecting piece at the top of the reaction tube contained a window for observing the susceptor and an entrance path for the $CH_3SiCl_3 + H_2$. The reaction tube was 45-millimeter-inside-diameter quartz. A pyrex connecting piece at the bottom of the reaction tube provided support for the susceptor and an exit path for the residual gases. At the exit, a glass check valve with a Teflon float prevented any backflow of air into the system.

The susceptors were vertical cylinders of spectroscopically pure graphite, 2.5 centimeters in diameter and 5 to 10 centimeters high. A typical susceptor design and temper-

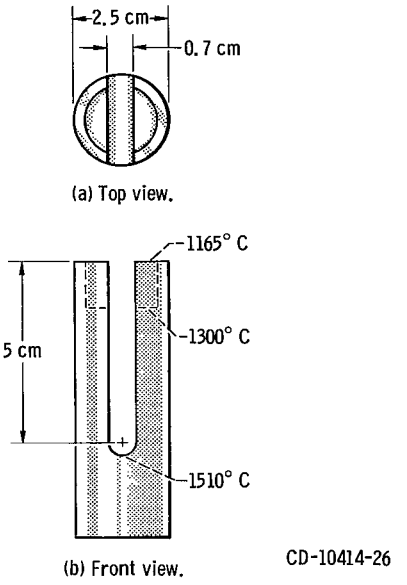

Figure 2. - Susceptor design and typical temperatures.

ature profile is shown in figure 2. The recessed rim at the top of the susceptor allowed observation of crystals growing in the slot. Without this recessed rim, the growth at the top of the susceptor blocked the view of the lower part of the slot, where crystals normally grew. Crystals also grew in a band around the outside of the susceptor. Before each run, the susceptors were outgassed overnight at the temperature of the particular crystal growth run. The outgassing temperature was limited by the quartz reaction tube, which was not normally water-cooled.

An automatic optical pyrometer provided both temperature measurement of the susceptor and observation of the crystals during growth. The sighting path was through a prism and pyrex window at the top of the reaction tube. Temperature errors due to the prism and window were determined by calibration against a platinum - platinum 13 percent rhodium thermocouple which was in a cavity in the susceptor. Normally, temperature measurements were made by sighting on the surfaces of the susceptor; hence, the temperatures reported herein are corrected for the prism and window, but not for emissivity. However, the errors in the temperature measurements are certainly less than $\pm 10^{\circ}$ C. The pyrometer also provided a signal for the temperature controller, which in turn usually produced control to better than $\pm 1^{\circ}$ C during a typical 24-hour run.

CRYSTAL GROWTH

Figure 3 shows the growth on a typical slotted susceptor. Two distinct areas of depo-

Figure 3. - Growth pattern on a slotted susceptor which has been sectioned. The left view shows the inside of the slot and the right view shows the outside of the susceptor.

sition can be seen, separated by an area relatively free of deposition. The upper deposition is a mixture of polycrystalline β SiC with up to 50 percent free silicon. The lower growth area usually contained both 2H and β SiC crystals. These crystals nearly always grew singly rather than in a cluster from a common site on the susceptor. Cluster-type growth was typical in previous 2H SiC growth (refs. 11, 13, and 14). Inside the slot, where the longer crystals grew, the bands of growth curved upward toward the center of the slot, as shown in figure 3.

The 2H SiC crystals grew up to 3.3 millimeters long and up to 0.3 millimeter in diameter. They were tapered and ended in either a point or a ball-shaped deposit of polycrystalline β SiC. Also, bands of β SiC often occurred around the outside of the 2H crystals, which were always hexagonal in cross section. The crystals were usually transparent although some opaque ones were found at the higher CH_3SiCl_3 concentrations. The color of the crystals depended somewhat on the growth conditions. They tended to be colorless at the lower CH_3SiCl_3 concentrations (about 0.4 molar percent) and amber colored at the higher concentrations (about 0.8 molar percent). Some selected crystals from one of the runs are shown in figure 4. A cross section of one of the larger crystals is shown in figure 5.

The β SiC crystals were polycrystalline and occurred in several shapes. Some were tapered and curved with a nearly round cross section. Others were straight with a rough

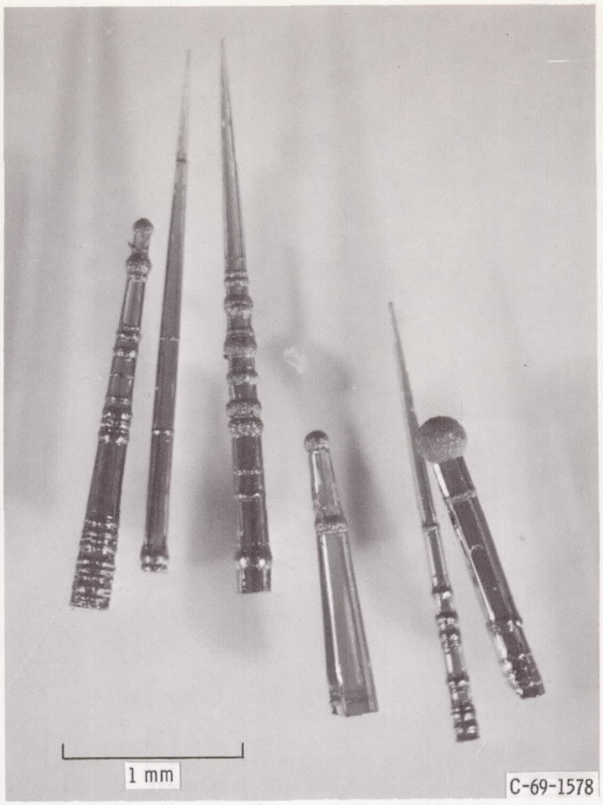

Figure 4. - Selected 2H silicon carbide crystals from single run.

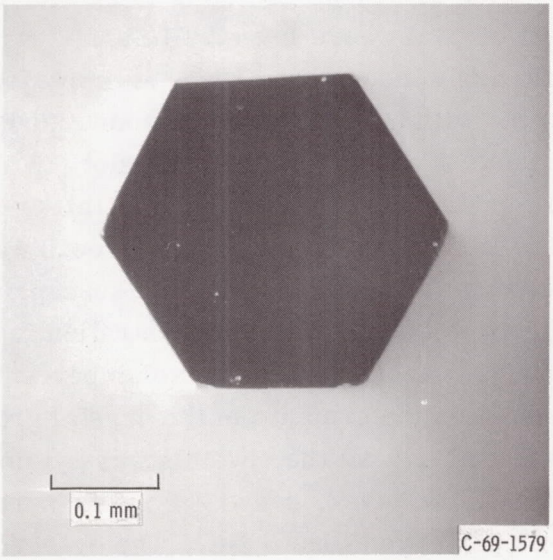

Figure 5. - Cross-sectional view of one of larger 2H silicon carbide crystals.

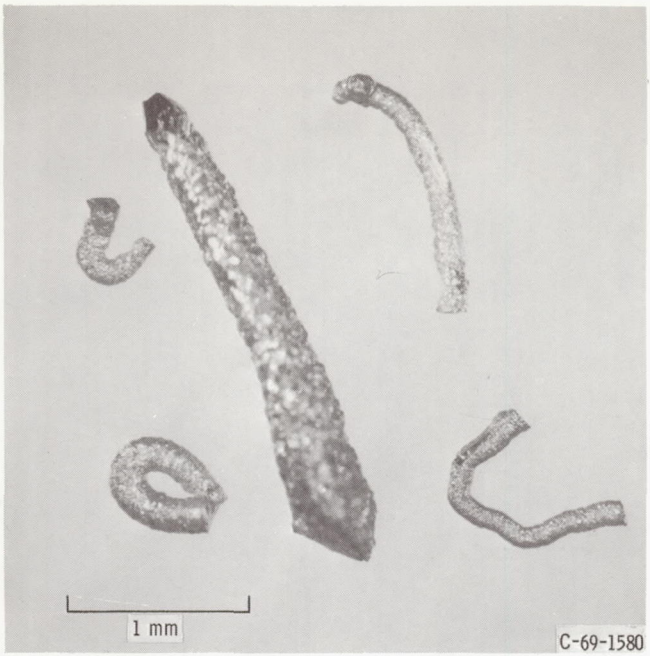

Figure 6. - Some typical β silicon carbide crystals.

star-like cross section. They were transparent and deep yellow in color. A few typical crystals are shown in figure 6.

All of the crystals with a hexagonal cross section normal to the long axis were strongly birefringent. These crystals exhibited extinction parallel to the long axis, indicating this axis to be the c-axis. X-ray photographs using the rotating crystal technique verified that the birefringent crystals were indeed 2H SiC.

The temperature profile along the susceptor was an important factor. The most successful 2H growth occurred with a temperature of about 1200° C at the top of the susceptor increasing to about 1500° C at the bottom of the slot. With this temperature profile the two areas of growth described at the beginning of this section occurred on the susceptor. The temperature in the area of the crystal growth was in the range 1300° to 1400° C. When the temperature was constant along the susceptor, no 2H crystals grew.

Another important factor was the CH_3SiCl_3 concentration in H_2. As the CH_3SiCl_3 concentration was increased from about 0.1 to 0.9 molar percent, the ratio of 2H SiC to β SiC crystals, which nucleated on the graphite in the crystal growth band, also increased. Below about 0.4 molar percent nearly all the crystals were yellow β SiC; above 0.8 molar percent nearly all were 2H SiC. However, above 0.8 molar percent the crystals were heavily overgrown with black polycrystalline β SiC. The overgrowth occurred as bands around the outside of the crystals, in addition to the ball-shaped deposits on the ends of the crystals. Examples of this overgrowth are shown in figure 4.

Visual observations of the crystals during growth showed that the VLS mechanism was not involved. Normally, the growing crystals at a temperature in the range 1300° to 1400° C were silhouetted against the background of the bottom of the susceptor slot at a temperature of about 1500° C. The optical system of the pyrometer was sufficient to allow resolution of objects smaller than 50 microns. It was observed that early in the growth, the 2H SiC crystals were thin and tapered to a point. The longer crystals maintained this same tapered shape throughout the growth. Those crystals that had a ball-shaped deposit on the end did not grow any further in the c-direction. If the VLS mechanism had been the dominant crystal growth mechanism in this case, the larger crystals would have had a stubby appearance early in the growth. The reason is that growth would have been only in the c-direction and not in the a-directions; thus, early in the growth the length to diameter ratio would have been small. Since this stubby shape was not observed at any time, the conclusion is reached that the VLS mechanism was not involved in this 2H SiC growth.

DISCUSSION

The successful 2H SiC crystal growth described in this report resulted from an effort to develop semiconducting devices for use at high temperatures. Since many devices require crystals larger than those grown so far, it is encouraging that 2H SiC crystals can be grown by some other mechanism than the VLS mechanism. Up to now only whisker-size crystals of any material have been grown by the VLS mechanism.

The role of impurities in the growth of 2H SiC is still in question. The recent paper by Knippenberg and Verspui (ref. 15) indicated that impurities are necessary in the growth of 2H SiC. The work by Ryan et al. (ref. 14) indicated that impurities could determine whether 2H or β SiC would nucleate. The work reported herein, however, indicates that proper control of susceptor temperature profile and CH_3SiCl_3 concentration can also determine whether the 2H or β polytype grows. Thus, the fact that impurities may not be necessary for the growth of 2H SiC is significant in that impurities (except for intentional dopants) are usually undesirable in materials to be used in the fabrication of semiconductor devices.

Some speculation can be made on the reason for the necessity of the susceptor temperature profile described in this report. An initial reaction of $CH_3SiCl_3 + H_2$ at about 1200° C takes place yielding (1) the SiC + Si deposit at the top of the susceptor and (2) some undetermined gaseous species probably rich in carbon. A second reaction involving these gaseous species takes place at 1300° to 1400° C, producing 2H SiC.

IMPORTANT RESULTS

This report describes preliminary results of an effort to determine the mechanism and the optimum conditions for the crystal growth of 2H SiC. Successful growth was achieved on an inductively heated graphite susceptor by the reduction of methyltrichlorosilane (CH_3SiCl_3) in a H_2 atmosphere. Visual observations of crystals during growth showed that the vapor-liquid-solid (VLS) mechanism was not involved. Important factors affecting the growth were the temperature profile along the susceptor and the CH_3SiCl_3 concentration in H_2.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, April 28, 1969,
120-27-01-25-22.

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